

Long-Term Immersion Testing of Alloy 22 and Titanium Grade 7 Double U-bend Specimens

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LONG-TERM IMMERSION TESTING OF ALLOY 22 AND TITANIUM GRADE 7 DOUBLE U-BEND SPECIMENS

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ABSTRACT

Double U-bend specimens of Alloy 22 (N06022) and Titanium Grade 7 (R52400) were exposed to a naturally aerated concentrated Basic Saturated Water (BSW) electrolyte at 105°C for over six years. Different type of discoloration of the Ti Gr 7 and Alloy 22 specimens was observed. General Corrosion was minimal and not distinguishable under a scanning electron microscope. None of the tested specimens suffered environmentally assisted cracking (EAC) or localized corrosion under the tested conditions. The specimens retained their residual stress after the long environmental exposure.

Keywords: Ti Gr 7, Alloy 22, Basic Saturated Water, Residual Stresses.

INTRODUCTION

Yucca Mountain is being studied as the site for a nuclear waste repository [1]. One of the reasons Yucca Mountain was selected is because it is a stable geologic formation that could contain the waste for thousand of years. Additionally, engineered barriers will be constructed to further isolate the waste from the environment. These engineered barriers include a double walled cylindrical container and a drip shield. The external wall of the container will be Alloy 22 (N06022) and the drip shield will be made mainly using Titanium (Ti) Grade 7 (R52400) [1]. Alloy 22 is nickel (Ni) based and contains approximately 22% chromium (Cr), 13% molybdenum (Mo),

3% tungsten (W) and 3% iron (Fe) [2]. Ti Gr 7 is over 99% Ti and contains approximately 0.15% palladium (Pd) [3].

Both Alloy 22 and Ti Gr 7 are extremely resistant to all forms of corrosion, including general corrosion (e.g. passive or uniform corrosion), localized corrosion (e.g. crevice and pitting corrosion) and environmentally assisted cracking (e.g. stress corrosion cracking and hydrogen embrittlement) [1,4]. The general corrosion rate of Alloy 22 after more than 5 years immersion in multi-ionic solutions simulating concentrated ground waters was extremely low (below 10 nano-meters per year) [4]. Similarly, Ti Gr 7 also showed low corrosion rates after 2.5 years exposure in simulated concentrated ground waters [4]. The highest reported corrosion rate for Ti Gr 7 was 46 nm/year measured in simulated concentrated water (SCW) at 90°C [4]. SCW, which is approximately 1000 times more concentrated than the ground water, and has a pH of 8-10 [4].

Alloy 22 and Ti Gr 7 are also highly resistant to localized corrosion. The critical crevice corrosion temperature for Alloy 22 and Ti Gr 7 in ferric chloride solutions (ASTM G 48) are 70°C and ~200°C respectively [5-7]. Electrochemical studies have shown that Alloy 22 had high crevice repassivation potentials and that the presence of nitrate and other anions inhibit the occurrence of crevice corrosion [8, 9]. Immersion studies at the free corrosion potential showed that both Alloy 22 and Ti Gr 7 U-bend specimens were free from cracking after

exposure for 5 and 2.5 years respectively to simulated concentrated ground waters from pH 2.8 to 10 [10, 11].

The aim of this study was to examine the corrosion behavior of Ti Gr 7 and Alloy 22 in basic saturated water (BSW) at 105°C using double U-bend specimens.

TEST SOLUTION

The ground water at the Yucca Mountain repository site is generally represented by the concentration of the J-13 well water [12]. J-13 is carbonate/bicarbonate rich and contains the cations sodium, potassium, calcium and magnesium [12]. J-13 is used to represent the composition of both the water in the saturated zone (significantly below the repository level) and of the water that is trapped in the unsaturated zone rock. If J-13 water is concentrated by evaporation by a factor of 1000, it becomes alkaline (pH 8-10) and contains only the cations sodium and potassium. The resulting brine of this evaporation is called simulated concentrated water (SCW) and it is also rich in carbonate/bicarbonate (70 g/L) and other anions such as sulfate (16.7 g/L), nitrate (6.4 g/L) and chloride (6.7 g/L).

The Basic Saturated Water (BSW) solution is highly alkaline and it was formulated in the laboratory to simulate extreme concentrating conditions of the J-13 water. There are two types of BSW water, one with carbonate and silicate called BSW-CS and another which is alkalinized with sodium hydroxide and does not contain carbonate or silicate. The latter solution is called BSW-13 because it has an as-prepared pH of 13. Carbonate and silicate are excluded from BSW-13 to avoid subsequent effects on pH. Table 1 shows the composition of the BSW-13 water used for the current tests. The major anions in this test solution are chloride, nitrate, and sulfate, and the major cations are sodium and potassium. The BSW-13 water contains approximately 32% by weight in dissolved salts (Table 1), while the original J-13 water contained only 0.03% in dissolved salts [12]. The pH of the BSW-13 solution is determined by the sodium hydroxide addition. The boiling point of the BSW-13 solution is approximately 109°C.

EXPERIMENTAL

Specimens

The Alloy 22 and Ti Gr 7 tested specimens were double Ubends, that is, they were composed of two self nesting strips of material (Figure 1). Each Ubend specimen is designed according to the guidelines in ASTM G 30. The double Ubend specimens are suitable for detecting at the same time environmentally assisted cracking (under constant deformation tensile stresses) and crevice corrosion in the occluded region between both strips and under the ceramic washers. The thickness of the Alloy 22 strips was 0.071 inch (1.8 mm) and the thickness of the Ti Gr 7 strips was 0.059 inch (1.5 mm). The internal strip specimen is the A-type and the external is the

B-type, that is, there is a crevice between the inner surface of the B specimen and the outer surface of the A specimen.

Table 1. Nominal Chemical Composition of the BSW-13 Testing Electrolyte

	Formula	Concentration (molal, m)		
KCl	8.7 g	1.77		
NaCl	7.9 g	2.05		
NaF	0.2 g	0.07		
NaNO ₃	13.6 g	2.42		
Na ₂ SO ₄ (anhydrous)	1.4 g	0.15		
DI Water	66 g			
10 N NaOH	2 mL			
Total	99.8 g			
pH = 13, [NO3]/[C1] = 0.63				

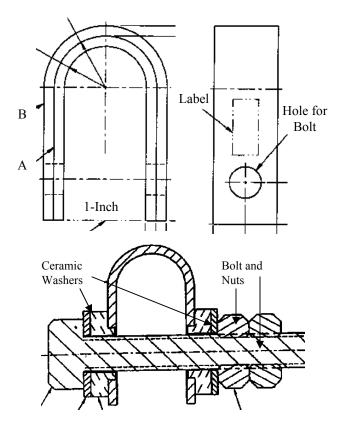


Figure 1. Alloy 22 and Ti Gr 7 Double U-bend Specimens

The heat and composition of the material used to fabricate the specimens is given in Table 2 (The first number in the chemical composition is the measured before the tests started, the second is the value provided by the alloy producer). The chemical composition of the material was measured by Laboratory Testing Inc. (LTI) (Pennsylvania). The mechanical properties and other metallurgical properties were also determined by LTI (Table 3). The mechanical properties listed in Table 3 are typical for Ti Gr 7 (ASTM B265) and Alloy 22 (ASTM B575). The double U-bend specimens were manufactured by Metals Samples (Alabama). They were mounted with the bolt and nuts so that the internal radius of curvature was 0.5 inch and the internal separation between the two legs of the A specimen (at the bolt location) was 1 inch (Figure 1). The specimens were electrically isolated from the bolt and nuts by ceramic washers or spacers.

Table 2. Chemical Composition of the Tested Double U-Bend Specimens

Material	Chemical Composition
Heat	Weight %
Manufacturer	
Ti Gr 7, ASTM B265	~99 Ti, 0.13-0.145 Pd,
Heat: R6940	0.11-0.135 O, 0.11-0.12
Titanium Industries Inc.	Fe, 0.008 N, 0.01-0.008
	С
Alloy 22, ASTM B575	~57 Ni, 22.0-21.3 Cr,
Heat: 2277-8-3203	13.6-13.08 Mo, 3.0-
Hastelloy C-22	2.93 W, 4.4-4 Fe, 2.3-
Haynes International	1.82 Co, 0.22-0.19 Mn,
Inc.	0.19-0.14 V, 0.05-0.026
	Si, <0.01-0.008 S,
	0.005-0.002 C

Table 3. Measured Mechanical Properties of the Material for the Double U-Bend Specimens

Properties	Ti Gr 7	Alloy 22
Yield Stress (0.2%) (ksi)	55	60
Tensile Strength (UTS) (ksi)	74	119
Elongation to rupture (%)	27	62
Hardness (RB)	-	92
ASTM Grain size	-	5.5

Testing Vessels and Procedure

Figure 2 shows the experimental setup. The Ti Gr 7 specimens were exposed in Vessel 1 (or Autoclave 1) and the Alloy 22 specimens were exposed in Vessel 2. The vessels were made using double walled glass. The solution in the

vessels was heated by oil circulating in the jacket between the double walled containers. The temperature of the oil was adjusted so that the temperature in the center of each vessel was 105° C. The temperature of the oil pumped in the jacket was generally in the vicinity of 125° C. At the beginning of the tests the aim of the temperature of the BSW solution was 109° C but it was later set to 105° C. The temperature was automatically recorded every hour. Figure 3 shows the temperature as a function of time for an approximately 2-week period in September 2000. In Figure 3 the average temperature in Vessel 1 (Ti Gr 7) was $106.92 \pm 0.37^{\circ}$ C and in Vessel 2 (Alloy 22) was $106.25 \pm 0.26^{\circ}$ C.

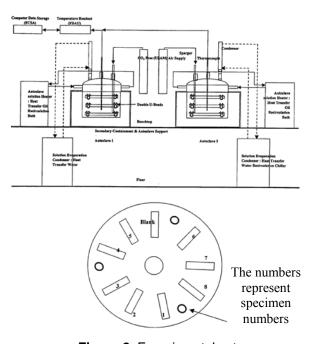


Figure 2. Experimental set-up

Air free of CO_2 was circulated in each vessel at a rate of 30-40 cc/min. The air exited the vessels through a condenser. The initial volume of BSW solution in each vessel was 5.7 liters. The solution level in each vessel was monitored and adjustments were made weekly using de-ionized water. In each vessel the specimens were suspended in three different levels or tiers. Initially, 20 specimens of each alloy were installed in each vessel. Figure 2 shows specimens 1-8 in the bottom tier. Specimens 9-16 were in the middle tier and specimens 17-20 were in the top tier.

Immersion Tests

The immersion tests for both vessels started on 14March2000. Vessel 1 was terminated on 21March2006 and Vessel 2 was terminated on 07August2006. Vessel 1 was terminated earlier due to a failure in the heating system. The

total exposure time for the Ti Gr 7 specimens in Vessel 1 was 2198 days (approx. 6 years) and the total exposure time for the Alloy 22 specimens in Vessel 2 was 2337 days (approx. 6 years 5 months) (Table 4). At the time the tests were terminated, of the 20 original specimens, only 17 Alloy 22 and 18 Ti Gr 7 specimens remained in Vessel 2 and Vessel 1, respectively. This is because on 26April2001, Specimen AARC22-20A/B was removed from Vessel 2 and transferred to the long term monitoring of the corrosion potential (Cell 4) [13]. Also, on 28August2001, Specimens ARC22-17A/B and ARC22-18A/B and Specimens ARTiGr7-17A/B and ARTiGr7-18A/B were removed from Vessels 2 and 1 respectively for an early inspection.

The initial pH of the electrolyte solution was set at 13. However, the measured pH value slightly decreased as a function of time. On 18May2001 and 02August2002, the pH of the solution in both Vessels 1 and 2 was measured and it was found to be between 10 and 11.

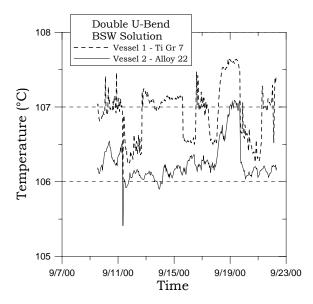


Figure 3. Hourly temperature vs. time in September 2000

RESULTS

Table 4 shows a list of the specimens removed from Vessels 1 and 2 at the end of the tests. All the specimens were covered with a thick layer of crusted white salt. The Ti specimens were black or exhibited a dark blue tint and the Alloy 22 specimens were shiny metallic, appearing to be plated. Figures 4 and 5 show the appearance of the specimens as removed from the vessels.

Table 4. Specimens Removed from Vessels 1 and 2

1		
	Vessel 1	Vessel 2
	Specimens	Specimens
	AR Ti Gr 7 U	AR C22 U
	1A-1B	1A-1B
	2A-2B	2A-2B
	3A-3B	3A-3B
	4A-4B	4A-4B
	5A-5B	5A-5B
	6A-6B	6A-6B
	7A-7B	7A-7B
	8A-8B	8A-8B
	9A-9B	9A-9B
	10A-10B	10A-10B
	11A-11B	11A-11B
	12A-12B	12A-12B
	13A-13B	13A-13B
	14A-14B	14A-14B
	15A-15B	15A-15B
	16A-16B	16A-16B
	19A-19B	19A-19B
	20A-20B	
	Total = 18	Total = 17
	Started: 14Mar00	Started: 14Mar00
	Ended: 21Mar06	Ended: 07Aug06
	Time: 2198 days	Time: 2337 days



Figure 4. Double U-bend Ti Gr 7 Specimen 9A/9B as removed from Vessel 1



Figure 5. Double U-bend Alloy 22 Specimen 9A/9B as removed from Vessel 2

Each one of the specimens listed in Table 4 was carefully examined for environmentally assisted cracking. None of the specimens listed in Table 4 suffered any type of cracking or other type of localized corrosion. It can also be argued that general corrosion was minimal as well. Titanium suffered staining by reaction with the environment but corrosion was not obvious. Similarly, it is possible that Alloy 22 suffered some type of general corrosion but the specimens did not appear corroded. It is likely that the plated appearance of the specimens was due to the re-deposition of nickel. Cross section analyses were not performed to determine if nickel has actually been deposited on the surface. General corrosion by weight loss could not be measured since the pre-immersion weight of the specimens was not recorded.

Disassembling of Three Specimens

Three Double-U Bend specimens of each material were washed with DI water and the bolt was removed for a complete inspection of the specimen. Even after washing for long time periods in water, some salt still remained on the specimen but this was not an impediment for the inspection. From Vessel 1, specimens AR Ti Gr 7 U 1A-1B, 10A-10B and 20A-20B and from Vessel 2 specimens AR C22 U 1A-1B, 10A-10B and 14A-14B were disassembled. Figures 6 and 7 show a detail of the apex of Ti Gr 7 and Alloy 22 1A and 1B specimens, respectively. None of the specimens showed environmentally assisted cracking. The Ti Gr 7 1A specimen showed a discoloration with a spot in the center. Scanning electron microscopy (SEM) confirmed that this was a thicker browner oxide but localized corrosion was not observed.

After removing the securing bolt, all six specimens showed a spring back phenomenon, clearly confirming that residual

stresses were present in the deformed material even after more than 6 years of exposure to the corroding electrolyte at 105°C. Table 5 shows the separation between the legs for each individual specimen after the holding bolt was removed. It is clear that Ti Gr 7 showed about 50% spring back from the original 1 inch internal separation between the legs of the A-type specimens. Similarly, Alloy 22 also showed spring back but in a slightly lower amount.

Table 5. Spring Back after Disassembling

Specimen	Distance (inches)	
AR Ti Gr 7 1A and 1B	1.503-1.613	
AR Ti Gr 7 10A and 10B	1.509-1.625	
AR Ti Gr 7 20A and 20B	1.507-1.628	
AR C22 1A and 1B	1.391-1.552	
AR C22 10A and 10B	1.380-1.633	
AR C22 14A and 14B	1.347-1.538	

Inspection for Crevice Corrosion

None of the disassembled double U-bend specimens showed any evidence for crevice corrosion between the Type A (inner) and Type B (outer) specimens. It is evident that Ti Gr 7 specimens showed a different discoloration at the point where the metal sheets touched (more brown) than at the points where a bigger gap may have existed (more blue) between the metal sheets A and B. Inspection in the SEM showed that the more brown area of contact between the Ti inner and outer specimens was due to the formation of thicker oxide, but localized corrosion was not found in this area. On the other hand, Alloy 22 was free of any discoloration that could be different from the plated-like surface observed before disassembling the specimens. Inspection for crevice corrosion was also conducted under the ceramic spacers or washers used to isolate the specimens from the bolt. No preferential attack was found under the crevice former. Figure 8 shows high magnification appearance of the surface of the Ti Gr 7 10B specimen of the surface outside and under the crevice former. In both cases the grinding marks of the specimen are visible. The grinding marks are clearer on the surface outside the crevice former. It is speculated that a thicker oxide film formed under the crevice former, slightly masking the sharpness of the grinding marks. For Alloy 22 1B there was less difference in the appearance of the surfaces outside the crevice former when compared to the surface under the crevice former (Figure 9). However it appears that the grinding marks are clearer under the crevice former than on the surface exposed to the bold solution. It is speculated

that a thicker film may have formed on the surface outside the crevice former.

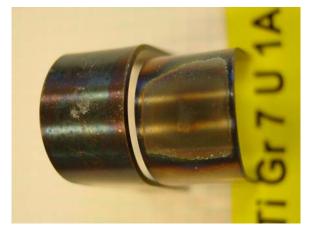
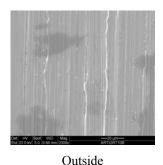


Figure 6. Ti Gr 7 Specimens 1A and 1B (left) after disassembling



Figure 7. Alloy 22 Specimens 1A and 1B (left) after disassembling



Under Crevice Former

Figure 8. Ti Gr 7 10B, X2000 Magnification

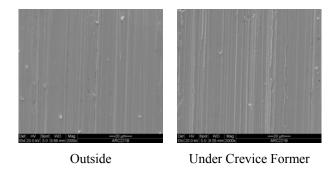


Figure 9. Alloy 22 1B, X2000 Magnification

SEM studies of the specimen type B surface at the edge of the ceramic washer also showed minimal corrosion. This was only evidenced in one out of three specimens examined for each alloy. Figure 10 shows the aspect of the attack for both Ti Gr 7 and Alloy 22 at the washer position interface. The attack is small in area and shallow since the grinding marks are still visible over the attacked areas.

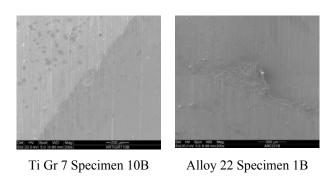


Figure 10. Ti Gr 7 10B and Alloy 22 1B, Magn. X200

DISCUSSION

Current results show that none of the specimens exposed to BSW-13 at 105°C for over six years suffered environmentally assisted cracking or localized corrosion under the tested conditions. Both Alloy 22 and Ti Gr 7 are highly resistant to all types of corrosion, general and localized. Data in the literature support the findings from the current long-term immersion tests. For example, slow strain rate tests of Alloy 22 in BSW-13 solution at 105°C did not show any signs of stress corrosion cracking, even at the highest polarization potentials of +400 mV SSC (saturated silver chloride) [14]. Andresen et al. used cyclic and constant loads to test compact tension Alloy 22 fatigue in pre-cracked specimens in BSW solution at 110°C

[15]. They tested both 20% cold worked and thermally aged specimens at a stress intensity level of 30 to 45 MPa \sqrt{m} and reported extremely low crack growth rates on the order of 2-8 x 10^{-13} m/s [15]. These values of environmental crack growth rates are in the limits of detection by the test equipment.

Andresen, et al. also tested Ti Gr 7 compact tensile specimens in the as-received and 20% cold-worked state. The specimens were loaded to 30 MPa \sqrt{m} in a BSW-12 solution at 110°C and subjected to very low cyclic loading (\sim 0.001 Hz). Crack length vs. time was monitored in-situ using a reversing direct current (DC) potential drop technique. The crack growth rate was approximately 1.3×10^{-11} m/s [15,16].

Crevice corrosion studies were conducted using electrochemical methods such as the cyclic potentiodynamic polarization on artificially creviced Alloy 22 specimens in BSW solution at 105°C. After forced polarization, shallow crevice corrosion was found but the repassivation potentials were high (on the order of 430 mV SSC) [17]. The corrosion potential ($E_{\rm corr}$) of Alloy 22 after 16 months immersion in BSW solution at 105°C was 27 and 46 mV SSC [13]. This $E_{\rm corr}$ is well below the region of anodic potentials (higher than 400 mV SSC) where Alloy 22 may be prone to crevice corrosion [13,17]. That is, it is unlikely that Alloy 22 would be naturally polarized in BSW solution to potentials where it may be susceptible to crevice corrosion.

Alloy 22 and Ti Gr 7 are also very resistant to general corrosion in BSW at 105° C. The general corrosion rate of Alloy 22 and Ti Gr 7 was measured using welded and non-welded coupons immersed in BSW-12 solution at 105° C for 8 weeks [18]. The corrosion rate of Ti Gr 7 was 0.022 mpy (~ 0.6 μ m/year) and the corrosion rate of Alloy 22 was 0.010 mpy $(0.25 \ \mu$ m/year) [18].

Published results show that both Alloy 22 and Ti Gr 7 are extremely resistant to all forms of corrosion. The fact that the currently reported results failed to show environmentally assisted cracking or generalized corrosion in both types of material confirms previously published results.

SUMMARY AND CONCLUSIONS

Double U-bend specimens of Alloy 22 and Ti Gr 7 (20 of each alloy) were exposed for over 6 years to simulated basic water (BSW-13) at 105°C

- None of the specimens suffered environmentally assisted cracking
- None of the specimens suffered localized corrosion between the two metal layers
- General corrosion was minimal for both types of materials.
 Minor corrosion has been observed at the washer/metal interface

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